EFFECTS OF PLASTIC CONSTRAINT ON THE CYCLIC AND STATIC FATIGUE BEHAVIOR OF METAL/CERAMIC LAYERED STRUCTURES

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Abstract

The role of metal layer thickness and resultant plastic constraint in the metal layer during the failure of metal/ceramic layered structures is examined under cyclic and static loading conditions. Crack growth experiments were conducted on sandwich specimens consisting of 99.999% pure aluminum layers bonded between 99.5 % pure polycrystalline alumina substrates while varying the layer thickness from 5 to 100 μ m. Under cyclic loading, crack growth occurred primarily at the interface separating the two materials, while cracks deviated into the alumina for thin layered samples at high driving forces. The toughness under critical loading increases with Al layer thickness, whereas the threshold driving force for growth in cyclic loading decreases with layer thickness. Under static loading in a moist environment, interfacial crack growth was never observed at measurable rates ($\geq 10^{-9}$ m/sec); however, for thin layered samples, subcritical cracks did deviate off the interface and grow, sometimes stably, into the alumina. Trends in growth rates and crack trajectories are examined in terms of the level of constraint, loading conditions, and environmental influences.

Keywords

Interface fracture, Fatigue crack growth, Stress corrosion cracking, Plastic constraint, Alumina, Aluminum

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1. Introduction

Metal/ceramic interfaces play a crucial role in dictating the mechanical properties of numerous reinforced and layered structures used in many engineering applications, including microelectronic packaging, multi-layered films, coatings, joints, and composite materials. The design of reliable engineering systems requires that the structural integrity of the interfaces be maintained during the lifetime of the component. Research focused on the strength and fast fracture behavior of metal/ceramic interfaces in metal/ceramic sandwich structures has been extensive; (Dalgleish et al., 1988; Dalgleish et al., 1989; Korn et al., 1992; Elssner et al., 1985; Evans and Lu, 1986; Evans et al., 1989; Evans and Dalgleish, 1992; Reimanis et al., 1991; Ritchie et al., 1993; Suo and Hutchinson, 1989; Turner and Evans, 1996; McNaney et al., 1994; Varias et al., 1991; Dalgleish et al., 1994) however, only a handful of studies have reported on the corresponding subcritical crack growth properties, specifically involving cyclic fatigue crack growth (Cannon et al., 1991; Shaw et al., 1994; McNaney et al., 1996; Gaudette et al., 1999; Peralta et al., 2000) and static fatigue by moisture-assisted cracking (Oh et al., 1987; Oh et al., 1988; Reimanis et al., 1990; Card et al., 1993).

The level of constraint of the plastic zone ahead of the crack tip imposed by surrounding elastic material is always a concern when applying fracture mechanics to monolithic, ductile materials. For example, the degree to which the plastic zone is constrained by the surrounding elastic material through the thickness of the sample (i.e., plane strain vs. plane stress conditions) is known to have a profound effect on fracture and fatigue properties. Additionally, loss of constraint can occur if plasticity extends to a free surface of a sample (e.g., the back face), again affecting the measured properties. In the case of a ductile layer between ceramic substrates, additional constraint of the plastic zone may occur when the plastic zone extends across the thickness of the metal layer, impinging on the ceramic (Figure 1). Constraint through the layer can thereby be affected by the metal layer thickness; indeed, theoretical investigations have predicted that the plastic zone size, the crack tip opening displacement, and the stress state near a crack tip are all affected by variations in the layer thickness (Varias et al., 1991; Hsai et al., 1994; Tvergaard et al., 1994; Mao and Li, 1999).

Despite the importance of this topic, there have been few experimental investigations on how failure mechanisms are affected by changes in plastic constraint, achieved through variations in the metal layer thickness (Ashby et al., 1989; Reimanis et al., 1991; Dalgleish et al.,

1994; McNaney et al., 1996; Dalgleish et al., 1989), with only the work of McNaney et al. (1996) focusing in part on fatigue failure. Trends of increasing strength with decreasing layer thickness in several metal/Al₂O₃ systems have been observed (Dalgleish et al., 1989, Dalgleish et al., 1994; Tomsia et al., 1995), whereas the corresponding variation in fracture toughness with layer thickness was less definitive. For a range of Al metal layer thicknesses studied (100 to 500 um), crack-tip blunting profiles indicated that the fracture toughness decreased significantly with decreasing layer thickness, although this trend could not be clearly demonstrated using conventional linear elastic fracture mechanics measurements due to excessive plasticity and possible loss of plastic constraint at the back face of the specimen (McNaney et al., 1996). An investigation using 10 to 100 µm thick layers in the gold/sapphire system, where thinner layers and lower toughness values limited plasticity in the gold, demonstrated a clear trend of decreasing toughness with decreasing layer thickness (Reimanis et al., 1991). As for fatigue failure, for the Al/Al₂O₃ system no effects of layer thickness have been previously observed on interfacial cyclic fatigue behavior for 100 to 500 µm thick layers, and additionally no static fatigue has been observed by moisture-assisted crack growth under static loading in this system (McNaney et al., 1996).

While it has been demonstrated in the aluminum/alumina system that strength increases with decreasing layer thickness, evidence indicates that long-crack fracture resistance properties follow an opposite trend; thus, the optimization of one property degrades the other and compromises must be made to achieve a suitable balance of strength and fracture toughness for a given application. For applications involving cyclic and/or static fatigue conditions, it is paramount that trends in fatigue properties with changing layer thickness, and the corresponding changes in plastic constraint, are also understood and incorporated into the design of systems for the best overall mechanical properties. Accordingly, the current paper is focused on investigating the Al/Al₂O₃ system using thinner layers than did previous investigations (5 – 100 µm) to explore the effects of increased constraint through the metal layer on both cyclic and static fatigue properties. In prior work, with 100 – 500 µm layers, it was found that cyclic loading led to interfacial failure, except when loads approach those for critical failure. The cracks either moved into the ceramic, becoming highly overdriven, or extended by the growth of microvoids in the aluminum that initiate at the interface (McNaney et al., 1996).

2. Experimental Procedures

Sandwich specimens with 5, 35, and 100 µm thick layers of 99.999% pure aluminum bonded between 99.5% pure polycrystalline Coors AD995 alumina were fabricated by liquid state bonding. The alumina microstructure has a bimodal grain size distribution, with grain sizes ranging from 3 to 30 µm, and a mean grain size of 13 µm. Blocks of alumina (21.3 mm square by 10.2 mm thick) were lapped flat to a 1 µm finish and carefully cleaned in acetone and ethanol before baking at 1000°C for 1 hr to remove organic impurities. High purity (99.999%) aluminum foils (100 and 35 µm thick layers) or evaporated coatings (5 µm thick layers) were placed between two alumina blocks and cold pressed to ~40 MPa before placing in a closed, high purity alumina crucible for bonding. Sandwich assemblies were heated to a temperature of 980°C in a gettered argon environment and held for 5 min before cooling. Further processing details are given elsewhere by Dalgleish et al. (1989). Bonded blocks were machined into 3 mm thick, 20 mm wide, compact tension, C(T), specimens for mechanical testing. Additionally, monolithic alumina C(T) samples were also machined for measurement of bulk alumina properties.

Cyclic fatigue-crack growth rates were measured in room air at 25°C (20 - 40% relative humidity) in general accordance with ASTM standard E647. Tests were conducted at a frequency of 25 Hz (sine wave) using computer controlled servo-hydraulic testing machines at a positive, constant load ratio (ratio of minimum to maximum loads) of R = 0.1. Crack lengths were continuously monitored by compliance methods using strain gauges mounted on the back face of the specimen, specifically on the alumina adjacent to the aluminum layer. Crack driving forces were assessed using the range of strain energy release rate, $\Delta G = G_{\text{max}} - G_{\text{min}}$. For the samples used in this study, where the layer thickness, h, is small compared to all other relevant sample dimensions, the strain energy release rate, G, is essentially unaffected by the presence of the layer (Suo and Hutchinson, 1989). G can be calculated from standard linear elastic stress intensity solutions for monolithic samples, K_I^{∞} , using the relationship:

$$G = \frac{K_I^{\infty^2}}{E'} \tag{1}$$

where E' = E in plane stress and $E/(1-v^2)$ in plane strain, E is Young's modulus for alumina, and v is Poisson's ratio. Fatigue crack growth rate versus applied driving force (i.e., $da/dN - \Delta G$)

curves were measured using both increasing and decreasing loading schemes (i.e., under increasing and decreasing ΔG conditions); the later method was used to obtain ΔG_{TH} fatigue thresholds, that were defined as the applied ΔG corresponding to growth rates below $\sim 10^{-10}$ m/cycle. Specifically, cracks were initiated by cycling until stable interface cracks formed ahead of the machined notches. Then, the loads were either incrementally reduced to measure the behavior approaching the threshold or increased to obtain the high velocity portion of the crack growth curves. The rate of change in loads was sufficiently slow to allow the crack blunting or bridging levels to adjust to the current load and level of G, i.e., the crack extension at each load level exceeded several plastic zone sizes.

After fatigue testing, fracture toughness tests were performed on the fatigue-cracked C(T) samples to assess trends in fracture toughness with changing layer thickness. The fracture toughness value was characterized in terms of the critical strain energy release rate, G_c , calculated from the peak load at failure using Eq. (1).

Moisture-assisted crack growth (static fatigue) experiments were conducted on fatigue pre-cracked C(T) specimens in a controlled high humidity (> 95% relative humidity) room air environment, where the fatigue pre-cracks initially were located at the interface. High humidity conditions were maintained by bubbling room air twice through distilled water and into a closed testing chamber. Samples were tested under constant load, i.e., with increasing crack driving forces. To determine crack velocity, da/dt, crack lengths, a, were monitored in situ using a back face strain to determine the unloading compliance curve. Additionally, crack lengths were periodically verified by the more accurate method of unloading the sample and measuring the actual unloading compliance. Samples were initially held at loads where reasonably measurable growth rates $(da/dt > 10^{-9} \text{ m/sec})$ could not be achieved. Loads were then step increased and held until measurable growth could be detected at some constant load, at which point all tests were conducted using an increasing loading scheme due to the increasing G field associated with constant loading for the C(T) geometry. When possible, samples were unloaded before critical fracture occurred.

3. Results

The results in Figures 2 – 5 show that cracks advanced along the Al/Al₂O₃ interface under cycling loading at low ΔG levels for all thicknesses examined here. At higher loads, the cracks

extended into the alumina. Once established in the alumina though, the cracks were unstable with the thicker metal layers as the driving forces markedly exceeded those to fracture the alumina. However, with the $5~\mu m$ Al layers, stable growth could be achieved for the cracks diverted into the ceramic under both cyclic and subcritical loading. As a consequence, R-curves and subcritical fracture properties were also measured in the alumina.

4. Discussion

4.1. Fracture Toughness Properties

A definitive trend of decreasing toughness with decreasing aluminum layer thickness, over the range of 5 – 100 μ m, was found for the fatigue pre-cracked Al₂O₃/Al/Al₂O₃ sandwich samples (Figure 2). For all samples tested, the fatigue pre-crack, which was located at the interface, was observed to initiate brittle failure in the alumina, with the Al₂O₃/Al interface remaining intact. Although fracture occurred in the alumina, fracture toughness values for the interface samples were well in excess of the initiation toughness of ~ 20 J/m² taken from the measured crack resistance curve (R-curve) for the bulk polycrystalline alumina (Figure 3). As fracture in the alumina depends on the stresses in the ceramic near the interface crack tip, the large increase in toughness may be attributed to stress reduction due to blunting of the crack tip into the aluminum.

Figure 4 shows direct evidence of this blunting phenomenon based on *in situ* images, taken in a field emission scanning electron microscope (FESEM), of a sandwich C(T) sample just prior to ultimate failure at 330 J/m². The tip of the fatigue crack at the interface of a 100 μ m thick layer sample was observed to blunt into the aluminum during loading from 73 to 267 J/m². The measured crack-tip opening displacement (CTOD) for this sample increased by permanent plastic deformation in the aluminum from sub-micron levels up to ~ 2.6 μ m at 267 J/m². Based on linear elastic fracture mechanics, local stresses near a blunted crack/notch, as in Figure 4, are considerably lower than those ahead of a sharp crack tip at the same applied driving force. In the case of the blunted interface cracks, a larger applied driving force is necessary to achieve the stresses necessary to initiate fracture of the alumina, thereby giving a higher measured fracture toughness for the sandwich specimens compared to that for bulk alumina.

The observed trend of increasing toughness with increasing layer thickness can also be attributed to greater crack tip deformation and blunting coupled with further effects of diminished plastic constraint through the thickness of the metal layer. To predict if plastic deformation is expected to impinge on the opposite alumina piece and become constrained, plastic zone size estimates¹ can be made for a crack at a bimaterial interface, where plasticity can extend into the aluminum unconstrained. Using the results of Shih and Asaro (1988), the distance, $r_{\rm pv}$, that the plastic zone extends into the aluminum, normal to the interface, can be predicted. Estimates reveal that the plastic zone would extend ~100 µm into the aluminum (i.e., comparable to the thickest layers of this study) at a driving force of only $\sim 11 \text{ J/m}^2$. This implies in all cases tested here that at driving forces necessary for fracture (> 70 J/m²), the plastic zone extends across the entire thickness of the aluminum layer, which limits the amount of plasticity and associated dissipation, and in turn the crack tip blunting, that can occur. For samples with thin layers, blunting is limited by the thickness of the metal layer at relatively low driving forces, leading to sharper crack tips compared to samples with thicker layers. This constraint also leads to higher stresses ahead of the crack tip; to illustrate this, the maximum mean, or hydrostatic, stress, defined as $\sigma_m = 1/3(\sigma_{11} + \sigma_{22} + \sigma_{33})$, normalized by yield stress, σ_0 , ahead of the crack tip can be used. Indeed, the computed maximum value of σ_m/σ_o ahead of the crack tip reaches a level of 6.65 at an applied driving force of $\sim 15 \text{ J/m}^2$ and $\sim 295 \text{ J/m}^2$ for 5 and 100 um thick layer samples, respectively. These values derive from computations of Varias et al., (1991) for the centerline crack configuration (Figure 1); it is expected that these predictions are quite reasonable for interface cracks where plasticity extends across the entire layer, and well ahead of the crack tip. Thus, it is implied that at the same applied driving force, samples with thinner, more highly constrained, layers experience higher local stresses ahead of the (sharper) crack-tip. Flaws in the alumina ahead of the interfacial crack tip can thus be triggered to failure and cause alumina fracture more easily for samples with thinner aluminum layers due to the more severe stress state.

Additionally, it should be noted that for the samples with 100 μ m, and perhaps 35 μ m, layer thicknesses, the G_c toughness values calculated from asymptotic stress intensity solutions may

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¹ A flow stress of 50 MPa for the aluminum was used for all plastic zone size calculations in this paper.

underestimate the total fracture energy due to excessive plasticity in the aluminum, i.e., from a violation of small scale yielding assumptions. Computed plastic zone size estimates in the direction ahead of the crack tip, $r_{\rm px}$, indicate that at a driving force of 280 J/m², the plastic zone already extends > 1 mm ahead of the crack tip for $h = 100~\mu m$ (Varias et al., 1991), which is not small compared to sample dimensions. Indeed, the work of McNaney et al. (1996) showed that for layer thicknesses $\geq 100~\mu m$, large scale plasticity with associated load point displacements accounts for a significant fraction of the fracture energy, and accordingly, no trend of decreasing fracture toughness with decreasing layer thickness could be determined when driving forces were calculated using linear elastic solutions. In the present study, however, this expected trend is observed since plasticity is limited in samples with 5 and 35 μ m thick layers such that small scale yielding assumptions are valid. Accordingly, it is expected that the trend seen in Figure 2 would be more pronounced if large scale plasticity were taken into account in the calculation of the crack driving forces for the samples with thicker layers.

4.2. Cyclic Fatigue Crack Growth Properties

4.2.1. Interfacial fatigue crack growth. An opposite trend of increasing crack-growth resistance with decreasing aluminum layer thickness was observed for cyclic fatigue of the Al₂O₃/Al/Al₂O₃ sandwich samples in the near-threshold regime (Figure 5), with crack propagation occurring exactly at the interface for all data shown. Samples with 5 μm thick layers showed a factor of two higher fatigue threshold compared to 100 μm thick layer samples, as well as an order of magnitude lower growth rates at comparable driving forces in the near threshold regime, while samples with 35 μm thick layers exhibited intermediate behavior in both regards. Data from McNaney et al. (1996), using samples with 100 to 500 μm thick layers where no such trend was observed, are also shown for comparison.

Evidence for the presence of fatigue striations on the aluminum side of the fracture surface was seen, similar to that observed by McNaney et al. (1996), suggesting a mechanism of fatigue crack propagation similar to that of ductile metals, with crack advance occurring by a process that involves blunting and re-sharpening of the crack tip, with individual striation markings corresponding to each blunting event. The amount of crack advance per cycle is typically directly related to the amount of blunting at the crack tip, with the cyclic growth increment

typically scaling with some fraction of the crack-tip opening displacement, i.e., ~ 0.1 to 0.3 for mode I self-similar growth in ductile metals (e.g., Gu and Ritchie (1999). Thus, if the plastic constraint restricts the degree of blunting during each cycle, this should act to reduce the growth rate for thinner layers. While this gives rise to higher fatigue resistance in the more highly constrained samples, it is important to note that for layer thicknesses ≥ 100 µm, the computed plastic zone width, $r_{\rm py}$, at the crack tip is small enough near the fatigue threshold (~ 35 µm at 4 J/m² (Shih and Asaro, 1988)) so that it does not extend completely across the metal layer. In this range of layer thicknesses, no effect of layer thickness is to be expected; indeed, experiments by McNaney et al. (1996) observed no layer thickness effects on fatigue thresholds and velocities under cyclic loading in the range of 100-500 µm thick aluminum layers.

Examination of the fatigue surface of 5 µm thick layer samples after failure revealed a crack path alternating between one interface and the other during fatigue crack growth (Figure 6a). While macroscopic jumps of the entire crack front from one interface to another were not uncommon for all samples, the additional localized jumps seen in Figure 6a, distributed across the fracture surface, were unique to 5 µm thick layer samples. Observations of crack profiles in unbroken samples (Figure 6b) revealed such jumping to be initiated predominantly at flaws in the alumina microstructure. In Figure 6b the crack, originally propagating on the upper interface, re-initiated on the other side of the layer at a flaw in the alumina; it then grew to, and propagated along, the lower interface. Crack jumping from interface to interface leads to bridging ligaments of aluminum in the crack wake, which may sustain some of the applied load and give rise to an increased in the measured fatigue threshold (Cannon et al., 1991); however, a recent study of the fatigue behavior of 2 µm thick layers of 99.999% aluminum between sapphire substrates, wherein smaller flaws exist and no crack jumping was reported, described a fatigue threshold of $\sim 10 \text{ J/m}^2$ (Gaudette et al., 1999). This result is similar to results for the 5 µm thick layer samples and significantly higher than the threshold of 4 J/m^2 found for 100 μ m thick layer samples in the present work, suggesting that effects on the fatigue threshold due to crack jumping are secondary to that of plastic constraint.

4.2.2. Fatigue crack deviation into the alumina. At higher driving forces, fatigue cracks leave the interface and grow into the alumina. Data for the stable crack growth in the alumina in the 5 µm thick layer samples are represented by open symbols in Figure 7. Crack initiation and

growth into the alumina for these samples is attributed to the higher local crack tip stresses due to plastic constraint through the metal layer than would occur for $h > 100 \, \mu m$. For the 5 μm thick Al layers, crack growth in the alumina initially occurs at driving forces lower than necessary for growth of large cracks in bulk polycrystalline alumina (Figure 7); data for the two cases only merges after $> 2 \, mm$ of crack growth. Many alumina alloys, including Coors AD995, have revealed a true cyclic fatigue effect, with degradation of grain bridges attributed as the mechanism for crack advance (Lathabai et al., 1991; Guiu et al., 1992; Dauskardt, 1993; Gilbert et al., 1995; Geraghty et al., 1999). Under monotonic loading, when intergranular fracture is predominant in alumina, frictional tractions along the fractured grain boundaries in the crack wake sustain load and shield the crack tip from part of the applied driving force, resulting in rising toughness with crack extension as these bridges develop (Swanson et al., 1987; Mai and Lawn, 1987; Bennison and Lawn, 1989). Evidence of this behavior can be seen in the rising R-curve shown in Figure 3. As the crack is cycled open and closed, however, the grain bridges that provide toughening in the alumina degrade by wear, thereby promoting crack advance.

After the crack leaves the interface and enters the alumina, crack growth occurs at higher growth rates than typically measured for bulk alumina for the first ~ 2 mm of growth. Since alumina demonstrates rising crack resistance with crack extension under monotonic loading (Figure 3), it is not surprising to observe crack size effects under cyclic loading as well. Crack size effects under cyclic loading are typically referred to as short or small crack² effects, with short cracks invariably growing at rates faster than long cracks at the same *applied* driving force (Ritchie and Yu, 1986). With ceramics, cracks may be considered to be short if the crack length is smaller than the distance over which extrinsic toughening (crack-tip shielding) takes place behind the crack tip. Crack size effects have been observed for small surface cracks in both alumina (Healy et al., 1997; Kishimoto et al., 1998), and other grain-bridging ceramics (Steffen et al., 1991; Zhang and Edwards, 1996; Gilbert et al., 2000), with these effects attributed to such extrinsic mechanisms as reduced grain bridging and compressive residual stresses.

While in the present case the interfacial cracks are physically long, alumina grain bridges can only begin to develop and sustain load after the crack has entered the alumina. The observed

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² Short or small fatigue cracks are defined as cracks small compared to: (i) the scale of microstructure, (ii) the extent of local inelasticity, or (iii) the extent of crack-tip shielding in the crack wake, where short cracks are small in one dimension and small cracks are small in all dimensions. They can generally grow at rates in excess of corresponding large cracks due to a higher local (near-tip) driving force experienced at the crack tip, e.g., (Suresh and Ritchie, 1984).

behavior is shown by the open symbols in Figure 7, where after the initial spike in growth rates corresponding to the change from an interfacial to alumina crack path, crack growth rates are then reduced before increasing again so the data merge with the standard large-crack fatigue data; this results in a v-shaped da/dN- ΔG curve, which is characteristic of short crack fatigue behavior observed in many extrinsically toughened materials (Andreasen et al., 1995; Kruzic et al., 1999; Gilbert et al., 2000). As the crack grows into the alumina, although the applied driving force is increasing, crack tip shielding due to the grain bridges initially increases at a faster rate, causing the effective driving force at the crack tip to decrease; this leads to an initial reduction in crack growth rates. After further crack growth, however, a steady state is reached where grain bridges are created and degraded at an equal rate, and the growth rates match those of the large cracks in the bulk alumina.

Bridging zones associated with cyclic fatigue crack growth in the alumina were observed in the present study to be on the order of 2 mm. Under monotonic loading, rising crack resistance is still observed after > 6 mm of crack growth (Figure 3), which suggests an equilibrium bridging zone greater than 6 mm in length. Clearly this is much greater than the 2 mm zone observed in fatigue, which supports the notion that it is indeed the degradation of the bridging zone under cyclic loading that is responsible for fatigue crack growth in alumina ceramics. Additional experimental evidence of smaller equilibrium bridging zones under cyclic, as compared to monotonic, loading has been reported for alumina by Hu and Mai (1992).

Furthermore, a rough estimate of the bridging zone length under monotonic loading can be made by noting that effective grain bridging occurs only up to the point behind the crack tip where the crack opening displacement is approximately 1/4 to 1/3 of the average grain size, $d_{\rm g}$ (Hay and White, 1993; Steinbrech et al., 1987). Using a simple hinge model for crack opening, the extent of the grain bridging zone can thus be estimated by calculating the point where the crack opening is approximately in this range. The opening displacement at the loadline, δ , can be calculated using:

$$\delta = \frac{P}{E'B}V(\frac{a}{W})\tag{2}$$

where P is the applied load, B and W are the specimen thickness and width, respectively, and the function V(a/W) is given by Saxena and Hudak (1978). While for an unbridged, linear elastic crack the axis of rotation is typically taken to be 0.2(W-a) ahead of the crack tip (Veerman and

Muller, 1972), experimental observations by Steinbrech et al. (1987) of alumina specimens suggest that grain bridging interactions across the crack faces draw the axis of rotation very near the crack tip. Thus, taking the hinge of rotation to be at the crack tip, and assuming a linear crack opening profile from the crack tip to the loadline, the rule of similar triangles can be used to determine the point where the crack opening is approximately $d_g/4 - d_g/3$. Using this method a grain bridging zone extending some 6 to 8 mm behind the crack tip under monotonic loading is predicted for the present Coors AD995 alumina, also clearly greater than the 2 mm bridging zone experimentally observed in fatigue. Using this model, the measured bridging zone length of ~2 mm for cyclic loading implies that effective grain bridges exist behind the crack tip up to the point where the crack opening is on the order of $d_g/12$, which may be a useful parameter for future predictions of bridging zone lengths under cyclic loading in grain bridging alumina ceramics.

In the present study, two distinct crack trajectories have been observed under cyclic loading for a crack initially at the Al/Al₂O₃ interface. In addition to the interfacial crack path, under conditions of high loading and plastic constraint, it has been found the cracks may leave the interface and propagate into the alumina. Additionally, due to crack size effects, initial crack propagation in the alumina for interface samples with thin metal layers occurs at driving forces lower than necessary for propagation of large cracks in bulk alumina. While thinner, more highly constrained, layers show improved fatigue properties in the near threshold regime, the change in fatigue mechanism from ductile fatigue at the interface to brittle fatigue in the alumina at higher driving forces, coupled with the short crack effects, means that the samples with thin layers have inferior fatigue resistance at high driving forces.

4.3. Moisture-Assisted Crack Growth Properties

For cracks in Al₂O₃/Al/Al₂O₃ sandwich samples under static loading in a moist environment (>95 % relative humidity), separation along the Al₂O₃/Al interface was not observed in any of the samples tested. Crack growth did occur, however, in samples with thinner aluminum layers when interfacial fatigue pre-cracks left the interface to propagate into the alumina. As shown in Figure 8, initial crack growth in the alumina for the 5 µm thick layer samples occurred at driving forces lower than necessary for equivalent growth in fatigue pre-cracked bulk alumina samples. For samples with 35 µm thick aluminum layers, analogous crack growth occurred in two of three

samples, but only at driving forces higher than necessary to crack bulk alumina. Due to the high driving forces necessary to initiate growth into the alumina in these samples, once the crack had grown a short distance from the plastically deforming aluminum layer, catastrophic fracture occurred, limiting the amount of subcritical crack growth data that could be obtained. It should be noted that this was a true time-dependent effect, with failure occurring only after several hours at constant load. Indeed, limited stable crack growth was sometimes actually measured before final failure, as shown in Figure 8. In the case of samples with 100 μ m thick layers (and one sample with a 35 μ m thick layer), no subcritical crack growth was observed at measurable rates ($\geq 10^{-9}$ m/sec.) up to driving forces of ~ 200 J/m². Thus, there is a range of driving forces in which subcritical extension may be observable or not, depending upon the size of the flaw being activated in the alumina.

As discussed above for the fracture toughness behavior, under moisture-assisted crack growth conditions, crack propagation only occurred by crack deviation off the interface into the alumina. Accordingly, as reasoned for the toughness results, the higher resistance to moisture-assisted crack propagation for samples with thicker aluminum layers is attributed to increased crack tip plasticity and blunting that lowers the local crack tip stresses relative to values necessary for the crack to leave the interface and enter the alumina. At 22 J/m², where growth was first measured for 5 μ m thick layer samples, the predicted σ_m/σ_0 ratio is greater than 7, while a sample with a 100 μ m thick layer would need to be loaded to > 300 J/m² to experience similar stress levels ahead of the crack (Varias et al., 1991). At such high load levels, only catastrophic fast fracture of the alumina is possible, and moisture-assisted crack growth may not occur except possibly to initiate crack extension into the ceramic.

Figure 9 shows direct evidence that moisture assisted crack growth did not occur at the interface, with fatigue markings evident up to the point where the crack deviated into the alumina. The observed crack growth for the interface specimens was primarily intergranular in the alumina, and appeared to be identical to that observed in previous studies on moisture-assisted crack growth in bulk alumina (Evans, 1972; Freiman et al., 1974). Initial moisture-assisted crack growth in the 5 µm thick layer samples occurred at driving forces lower than for crack growth in fatigue pre-cracked bulk alumina samples. It should be noted, however, that the curve shown for the bulk alumina is not unique, and only represents data from the initial growth from large (several millimeters) fatigue pre-cracks. Subsequent measurements of crack velocity

(v-G) curves result in a shift of the data to higher driving forces, as illustrated in Figure 10. Two more v-G curves, measured in succession on the same sample at $\sim 40\%$ relative humidity, are shown in Figure 10, where each measured curve is successively shifted to higher driving forces. This effect has been observed previously in alumina by Steinbrech et al. (1983), with the shifting of the v-G data attributed to the systematic progression up the R-curve. Indeed, if the contribution of crack shielding (e.g., grain bridging) could be accounted for, a v-G curve for the crack tip could be considered unique or intrinsic to the material (Lawn, 1993). Alternatively, an asymptotic curve for long crack behavior could be developed, as was discussed for growth under cyclic loading.

The marked shift of the v-G data to lower driving forces for the 5 μ m thick layer samples indicates that there was a lower initial starting point on the alumina R-curve than for the fatigue pre-cracked bulk alumina samples, where initial pre-crack lengths were several millimeters in length. Indeed, initial crack growth for the 5 μ m layer samples occurred at a driving force of 22 J/m², which is very near the extrapolated initiation toughness of ~20 J/m² taken from the R-curve shown in Figure 3, where the fatigue pre-crack was only 230 μ m in length. The low driving force necessary for initial growth into the alumina for the 5 μ m thick layer samples implies an initial position near the beginning, or lowest point, of the alumina R-curve. As was found in cyclic fatigue, the results indicate a crack size effect for globally large interface cracks that only begin to develop grain bridging, and subsequent R-curve toughening, after they deviate off the interface into the alumina.

4. Conclusions

Based on an experimental study to investigate the role of metal layer thickness (over the range from 5 to 100 μ m) for the ceramic-metal Al₂O₃/Al/Al₂O₃ layered system, the following conclusions can be made:

1. The fracture toughness of the layered samples was seen to increase with increasing aluminum layer thickness. As the initial interfacial pre-cracks resulted in fracture into the alumina, this trend was rationalized in terms of enhanced crack tip deformation and blunting in the thicker metal layers (where plastic deformation is less constrained), which also lowered the local crack tip stresses relative to those needed to trigger cracking at flaws in the alumina.

- 2. The interfacial fatigue-crack growth resistance was found to decrease with increasing aluminum layer thickness at lower driving forces; this was observed in the form of lower fatigue thresholds and higher growth rates in the near threshold regime. Stable cyclic fatigue-crack growth occurred predominantly at the Al₂O₃/Al interface, except at higher driving forces. For the 100 μm thick layer samples, estimates of the plastic-zone size suggest that plasticity does not extend through the thickness of the layer at the fatigue threshold; for the 5 μm thick layer samples, conversely, the plasticity is constrained by the thickness of the layer, thereby limiting the degree of crack blunting and consequently the crack advance per cycle even at threshold.
- 3. At higher driving forces, the trajectory of the cyclic fatigue cracks was found to change, specifically from an interfacial path to crack growth in the alumina, resulting in substantially less crack growth resistance. For thicker layers, the growth in the alumina was unstable owing to the high *G* levels needed to trigger the transition. For 5 µm thick layer samples, stable growth occurred in the ceramic. Additionally, "short crack effects", attributed to lack of grain bridging for a crack just entering the alumina, caused the initial cracking to occur at lower driving forces than normally measured for large cracks in bulk alumina.
- 4. Under static loads in a moist environment, cracks in samples with 5 μm thick metal layers also left the interface and propagated sub-critically in the alumina; this did not occur in samples with 100 μm thick metal layers due to enhanced crack tip blunting in the aluminum. Due to the lack of an equilibrium crack-tip shielding zone of grain bridges in the crack wake, such initial crack growth into the alumina occurred at driving forces lower than that measured for crack growth from large fatigue pre-cracks in bulk alumina.
- 5. Whereas previous work has demonstrated that thinner metallic layers in alumina/aluminum structures produces higher strength ceramic/metal joints, the current study shows that although interfacial fatigue thresholds may also be improved, flaw tolerance is essentially reduced by lowering the overall fracture toughness and promoting subcritical crack growth off the interface and into the alumina. The latter phenomenon is observed under cyclic loading at high driving forces and under static loading in a moist environment, as well as in critical fracture.

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Figure Captions

- Figure 1: Schematic illustrating constraint of the plastic zone ahead of a crack tip and extending through the thickness of a metal layer bonded between two ceramic substrates.
- Figure 2: Fracture toughness values results for polycrystalline alumina/aluminum sandwich specimens as a function of metal layer thickness. All samples failed in the alumina.
- Figure 3: R-curve measured for Coors AD995 alumina demonstrating rising crack growth resistance with crack extension. Data shown were collected from a fatigue precracked C(T) sample where the pre-crack was grown only 230 mm from the machined notch to minimize the bridging that was generated during initial crack formation.
- Figure 4: Blunting of an interfacial crack into a 100 µm thick aluminum layer during loading *in situ* in the FESEM as the applied driving force was increased from 73 to 267 J/m². The sample failed at an applied driving force of 330 J/m² in the adjoining alumina.
- Figure 5: Fatigue crack growth results for interfacial cracks showing a decrease in crack growth rates with decreasing layer thickness in the near threshold regime. Additional data from McNaney et al. (1996).
- Figure 6: Evidence of crack jumping both (a) on the fatigue fracture surface and (b) in profile on a 5 mm thick layer sample. In (a) aluminum is seen in lighter contrast. Crack jumping is observed to initiate at flaws in the alumina. Direction of crack growth was from left to right.
- Figure 7: Fatigue crack growth rate results for a fatigue crack leaving the interface of a 5 μ m thick layer sample and growing stably in the alumina. Growth rates were initially faster than that in bulk alumina, with the data merging only after > 2 mm of crack growth.
- Figure 8: Moisture-assisted crack growth rates for interface and bulk alumina specimens in moist air (>95% relative humidity). Crack growth was in the alumina for all cases where discernible cracking occurred in an interface sample.
- Figure 9: Evidence of fatigue striations up to the point where the crack left the bimaterial interface during moisture-assisted crack growth. Direction of crack propagation was from left to right.
- Figure 10: Two sequentially measured *v-G* curves for a fatigue pre-cracked bulk alumina sample measured in 40% relative humidity room air. The curve measured second is shifted to higher driving forces, corresponding to starting at a higher portion on the alumina R-curve.